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19. ABSTRACT (Continue on reverse if necessary and identify by block number) Unsymmetrical 2,3-dimethyl-5-silaspiro[4,4]nona-2,7-diene (I) has been prepared by a dissolving metal reaction between 1,1-dichloro-1-silacyclopent-3-ene (III) and 2,3-dimethyl-1,3-butadiene. Ring opening reactions of I catalyzed by alkylolithium reagents and hexamethylphosphoramide (HMPA) have been explored. High yields of the dimer 2,3,12,13-tetramethyl-5,10-disiladispiro[4,4,4,4]octadeca-2,7,12,16-tetraene (II) are formed in these reactions. The structure of II in space group $P_1$ (triclinic) with unit cell parameters: $a=7.826(3)$ Å, $b=9.415(3)$ Å, $c=7.421(3)$ Å, $\alpha = 94.23^\circ$ , $\beta = 114.56(3)^\circ$ , $\gamma = 89.34(3)^\circ$ , $V=496.0(3)$ Å <sup>3</sup> , and $Z=1$ .			
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## APPENDIX VII

### Synthesis and Dimerization 2,3-Dimethyl-5-Silaspiro[4,4]nona-2,7-diene.

Young Tae Park, Stephen Q. Zhou, Dong Zhao, Georges Manuel, Robert Bau and William P. Weber\*

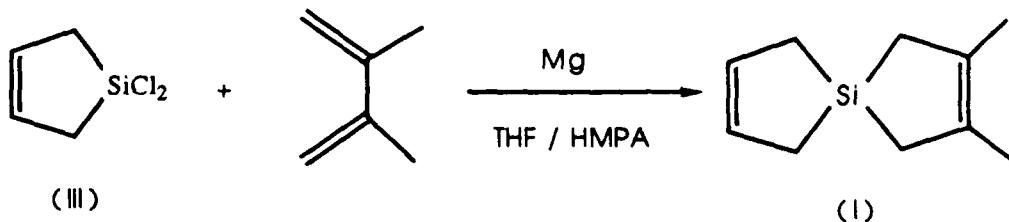
Donald P. and Katherine B. Loker Hydrocarbon Research Institute, Department of Chemistry, University of Southern California, Los Angeles, California 90089-1661 USA.

#### Summary:

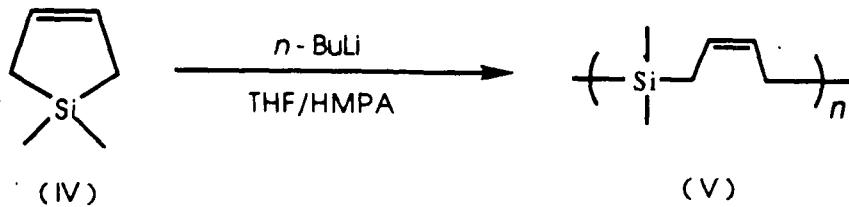
Unsymmetrical 2,3-dimethyl-5-silaspiro[4,4]nona-2,7-diene (I) has been prepared by a dissolving metal reaction between 1,1-dichloro-1-silacyclopent-3-ene (III) and 2,3-dimethyl-1,3-butadiene. Ring opening reactions of I catalyzed by alkyl-lithium reagents and hexamethylphosphoramide (HMPA) have been explored. High yields of the dimer 2,3,12,13-tetramethyl-5,10-disiladispiro[4,4,4,4]octadeca-2,7,12,16-tetraene (II) are formed in these reactions. The structure of II has been determined by X-ray crystallography. II crystallized in space group  $P\bar{1}$  (triclinic) with unit cell parameters:  $a = 7.826(3) \text{ \AA}^0$ ,  $b = 9.415(3) \text{ \AA}^0$ ,  $c = 7.421(3) \text{ \AA}^0$ ,  $\alpha = 94.11(3)^\circ$ ,  $\beta = 114.56(3)^\circ$ ,  $\gamma = 89.34(3)^\circ$ ,  $V = 496.0(3) \text{ \AA}^3$ , and  $Z = 1$ .

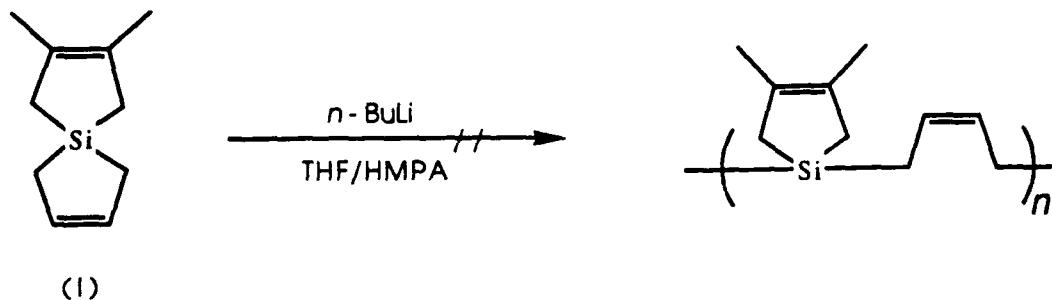
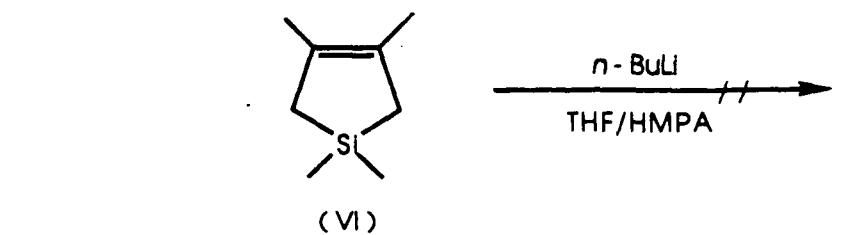
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While a few symmetrical 5-silaspiro[4,4]nona-2,7-dienes have been prepared,<sup>1-3</sup> no unsymmetrically substituted examples of this ring system are known. We have prepared unsymmetrical 2,3-dimethyl-5-silaspiro[4,4]nona-2,7-diene (I) by the dissolving metal reaction of 2,3-dimethyl-1,3-butadiene with 1,1-dichlorosilacyclopent-3-ene (III) and magnesium in THF and HMPA. The recently reported preparation of III from readily available starting materials makes this synthetic route feasible.<sup>4</sup>



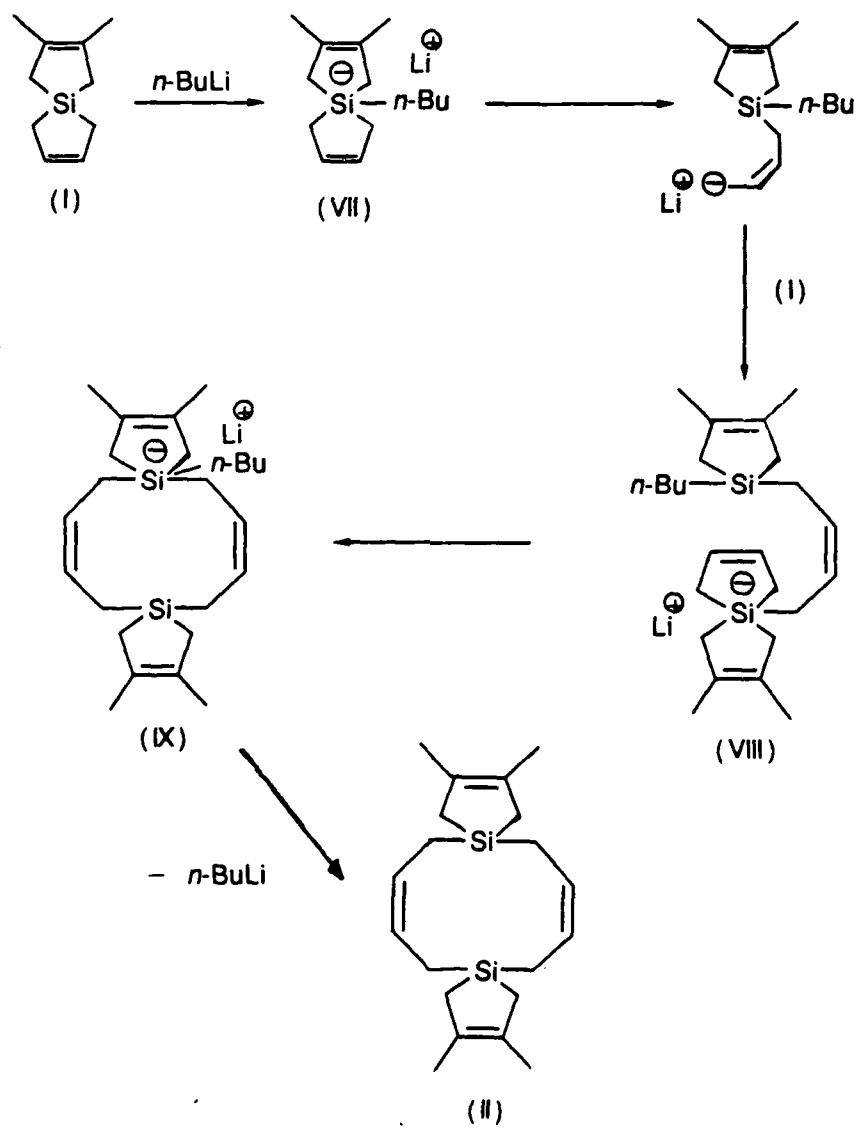
We anticipated that treatment of I with catalytic amounts of alkylolithium reagent and HMPA in THF at low temperature would result in a polymer formed by the selective ring opening of the unsubstituted 1-silacyclopent-3-ene ring of I. Such a polymer, poly(3,4-dimethyl-1-silacyclopent-3-en-1-ylene-*cis*-but-2-en-1,4-ylene), would have 1,4-(*cis*-but-2-ene) units bonded to the silicon atoms of the 3,4-dimethyl-1-silacyclopent-3-ene rings. This expectation was based on our previous work which has shown that while 1,1-dimethyl-1-silacyclopent-ene (IV) undergoes polymerization under these conditions to yield poly-(1,1-dimethyl-1-sila-*cis*-pent-3-ene) (V),<sup>5</sup> 1,1,3,3-tetramethyl-1-silacyclopent-3-ene (VI) does not.<sup>6</sup>





To our surprise, treatment of I under these conditions lead to almost quantitative formation of a dimer, 2,3,12,13-tetramethyl-5,10-disiladispiro[4,4,4,4]octadeca-2,7,12,16-tetraene (II). The central ring of II is the first example of the 1,6-disiladispiro-3,8-diene ring system of which we are aware. We believe that II is formed by anionic attack on the silyl center of I by an alkyl lithium reagent to form a pentacoordinate hypervalent silicon species (VII). Ring opening of the unsubstituted ring of VII occurs to generate a *cis*-allyl anion which reacts with another molecule of I to form a new hypervalent siliconate intermediate (VIII). This process must occur faster than rotation about the partial carbon-carbon double bonds of the *cis*-allyl anion which would convert it into a *trans*-allyl anion. This appears reasonably since an energy barriers of approximately fifteen kcal/mol have been measured for this type of isomerization process.<sup>7</sup> Ring opening of the unsubstituted five membered ring of

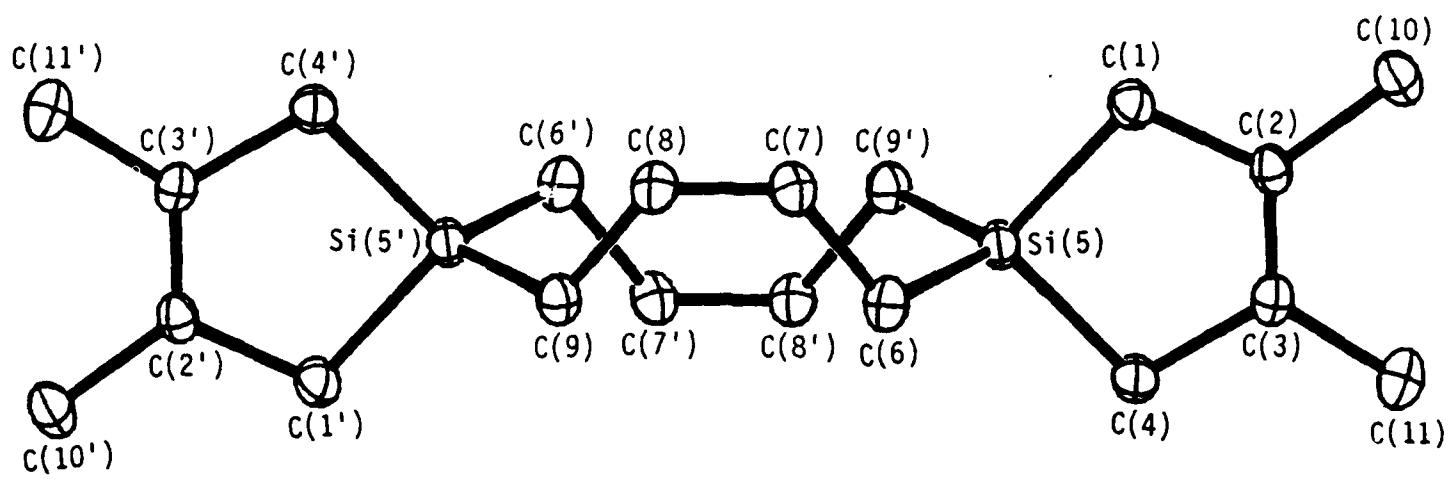
VIII leads to a new *cis*-allyl anion intermediate which reacts intramolecularly with the other silicon atom of VIII faster than it reacts with another molecule of I. This process forms the central ten membered ring of II and yields a new hypervalent siliconate species (IX) which loses a molecule of alkyllithium to give II. Thus alkyllithium reagents catalyze the conversion of I to II. This is unexpected since it is dependent on the ability of an allyl anion to displace an alkyllithium from a silyl center. This is unusual since allyl anions are thermodynamically more stable than alkyl anions.<sup>8</sup>



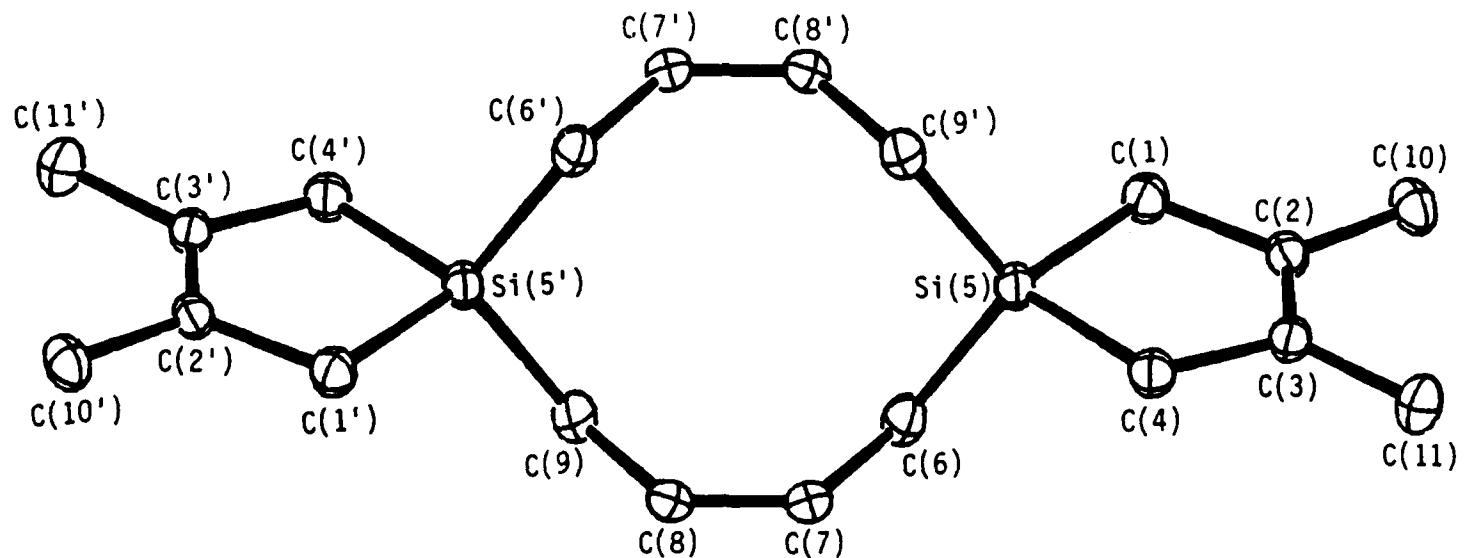
It was not possible to definitely assign the structure of II on the basis of NMR spectroscopy. Thus,  $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{29}\text{Si}$  NMR spectra were consistent with two highly symmetrical structures: II or 7,8,16,17-tetramethyl-5,10-disiladispiro[4,4,4,4]octadeca-2,7,12,16-tetraene. The chemical shifts of the vinyl protons and carbons favor II. In particular, the  $^1\text{H}$  NMR signals for the vinyl protons of IV are found at 5.73 ppm while those for V are found at 5.29 ppm. Similarly, the  $^1\text{H}$  NMR resonance for the vinyl protons of I comes at 5.91 ppm while those for II are found at 5.30 ppm. The  $^{13}\text{C}$  NMR signals assigned to the vinyl carbons of II are found at 130.58 and 122.92 ppm. These resonances can be compared to the vinyl carbon resonances for IV at 130.74 ppm and those for V which is found at 123.22 ppm. The  $^{13}\text{C}$  NMR signals assigned to the non-equivalent vinyl carbons of I are found at 131.16 and 130.87 ppm. Similarly, the vinyl carbon resonances of VI is found at 130.56 ppm. Finally, the  $^{29}\text{Si}$  resonance in I is found at 25.54 ppm while that of the dimer II is observed at 11.87 ppm. This upfield shift is similar in magnitude to that observed when one compares the  $^{29}\text{Si}$  NMR of IV at 16.5 ppm to that of V which is found at 2.17 ppm.

The structure of the dimer was confirmed by X-ray crystallography. The Si-C bonds of II are between 1.872 and 1.878  $\text{\AA}^\circ$ , while the C-C single bonds are between 1.495 and 1.516  $\text{\AA}^\circ$  in length. The C-C double bonds are between 1.325 and 1.327  $\text{\AA}^\circ$ . The C-Si-C bond angle in the five member silacyclopent-3-ene ring is  $95.2^\circ$ . For comparison this is slightly larger than the C-Si-C bond angle of  $92.6^\circ$  which is found in 1,1-dimethyl-2,3,4,5-tetra-

phenylsilole. 9,10



ORTEP diagram - side view of II.



ORTEP diagram - top view of II.

## Experimental

### X-ray Structure Analysis of II.

Crystals suitable for an x-ray structure analysis were grown by cooling a concentrated THF solution of II in a refrigerator. A crystal of dimensions 0.5 x 0.3 x 0.2 mm was used for crystal and intensity data collection.

X-ray data were collected at room temperature by using a Nicolet/Syntex P2<sub>1</sub> diffractometer with MoK<sub>α</sub> radiation and a maximum 2θ of 50°. The orientation matrix and unit cell parameters were determined from the angular setting of 15 well-centered reflections. Three check reflections showed no significant change in intensity during the period of data collection. A total of 1809 reflections were measured.

Compound II crystallizes in space group P<sub>1</sub> (triclinic) with unit cell parameters: a = 7.826(3) Å<sup>o</sup>, b = 9.415(3) Å<sup>o</sup>, c = 7.421(3) Å<sup>o</sup>, α = 94.11(3)°, β = 114.56(3)°, γ = 89.34(3)°, and V = 496.0(3) Å<sup>o</sup><sup>3</sup>. Application of direct methods<sup>11</sup> yielded the position of the silicon atoms; the other non-hydrogen atoms were located from a series of structure-factor-calculation/difference Fourier calculations. Full matrix least squares refinement<sup>11</sup> (including calculated positions of hydrogen atoms) yielded a final agreement factor of 3.3% for 1478 non-zero reflections [I > 3σ(I)]. For details of the X-ray structure and its determination see Supplemental material.

<sup>1</sup>H, <sup>13</sup>C and <sup>29</sup>Si NMR spectra were recorded on an IBM-Brucker 270-SY or Brucker AM-360 spectrometer operating in the Fourier

Transform mode.  $^{13}\text{C}$  NMR spectra were run with broad band proton decoupling. A DEPT pulse sequence was used to obtain  $^{29}\text{Si}$  NMR spectra. This was effective since all the silicon atoms have at least one methylene group bonded to them.  $^{12}$  Identical  $^{29}\text{Si}$  NMR spectra could be obtained by use of a heteronuclear gated decoupling pulse sequence (NONOE) with a pulse delay of 30 seconds.  $^{13}$  Ten to fifteen percent solutions in chloroform-d were used to obtain  $^{13}\text{C}$  and  $^{29}\text{Si}$  NMR spectra. Five percent solutions were used to obtain  $^1\text{H}$  NMR spectra. Chloroform was utilized as an internal standard for  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra.  $^{29}\text{Si}$  NMR spectra were externally referenced to TMS.

IR spectra were recorded on a Perkin-Elmer PE-281 spectrometer. Spectra of oils were taken as neat films on NaCl plates. IR spectra of solids were taken on KBr pellets.

Low resolution mass spectra were obtained on a Finnigan Mat Incos 50 GCMS instrument at an ionizing voltage of 70 eV. A 0.25 mm x 30 m fused silica DB-5 capillary column was used in the gas chromatographic inlet of the mass spectrometer. High resolution mass spectra were obtained at the University of California Riverside Mass Spectrometry Facility on a VG-7070 EHF mass spectrometer at an ionizing voltage of 20 eV. Exact masses were determined by peak matching against known masses of perfluorokerosene.

Elemental analysis was performed by Galbraith Laboratories, Knoxville, TN.

Tetrahydrofuran (THF) was distilled immediately prior to use from a deep blue solution of sodium benzophenone ketyl. Hexamethylphosphoramide (HMPA) was distilled from calcium hydride and was stored over activated 4  $\text{A}^\circ$  molecular sieves. 2,3-Dimethyl-1,3-

butadiene and active magnesium powder were purchased from Aldrich Chemical Co. Inc.

All glassware was dried overnight in an oven at 120°C. It was assembled and was flame dried under an atmosphere of purified Argon. All reactions and transfers were conducted under an atmosphere of purified Argon.

### 2,3-Dimethyl-5-silaspiro[4,4]nona-2,7-diene (II)

In a 500 mL three neck rb flask equipped with a reflux condenser, a pressure equalizing addition funnel and a Teflon covered magnetic stirring bar was placed magnesium powder (2.48 g, 0.1 mol), 2,3-dimethyl-1,3-butadiene (8.4 g, 0.1 mol), THF (63 mL) and HMPA (17 mL). <sup>14</sup> The flask and its contents were cooled to 0°C. I (10.4 g, 68 mmol) <sup>4</sup> and THF (20 mL) were placed in the addition funnel. This solution was added dropwise to the vigorously stirred suspension of magnesium powder over 1 h. The reaction mixture was allowed to warm to rt and was stirred for 72 h. Pentane (100 mL) was added. Saturated aqueous ammonium chloride (100 mL) was then added dropwise with vigorous stirring. The organic layer was separated, washed with water, dried over anhydrous magnesium sulfate and filtered. The organic solvents were removed by fractional distillation through a 15 cm vacuum jacketed Vigreux column. The residue was transferred to a smaller flask and the distillation was continued under reduced pressure. A fraction, bp 108-110°C/11 mm, 4.4 g, 40% yield was obtained. It had the following properties. <sup>1</sup>H NMR δ: 1.45(d,4H, J = 1.0 Hz), 1.51(d,4H, J = 1.1 Hz), 1.72(t,6H, J = 1.0 Hz), 5.91(t,2H, J = 1.0 Hz). <sup>13</sup>C NMR δ: 16.54, 19.18, 24.13, 130.87, 131.16. <sup>29</sup>Si NMR δ

: 25.54. IR  $\nu$ : 3020, 2970, 2890, 2880, 1605, 1440, 1395, 1205, 1175, 1090, 940, 820, 760, 725, 615  $\text{cm}^{-1}$ . GC/MS  $m/e$  (rel. intensity): 166(3.5), 165(12.9), 164(82.4)  $M^{+}$ , 149(4.0)  $M-15^{+}$ , 136(4.4), 123(3.2), 122(10.0), 112(3.7), 111(12.6), 110(100.0)  $M-\text{C}_4\text{H}_6^{+}$ , 97(4.0), 96(9.8), 95(83.2)  $M-\text{C}_4\text{H}_6-15^{+}$ , 84(2.1), 82(25.6)  $M-\text{C}_6\text{H}_{10}^{+}$ , 71.(2.2), 70(3.7), 69(17.2), 68(8.9), 67(28.3), 66(5.8), 65(2.7), 59(1.7), 58(1.8), 57(3.5), 56(5.3), 55(34.7), 54(16.5), 53(26.8), 51(2.5), 50(1.1). High resolution MS  $m/e$  Calcd for  $\text{C}_{10}\text{H}_{16}\text{Si}$   $M^{+}$ : 164.1021; Found 164.1023. Elemental Anal. Calcd. for  $\text{C}_{10}\text{H}_{16}\text{Si}$ : C, 73.09; H, 9.82. Found: C, 72.59; H, 10.20.

#### Reaction of I with *n*-Butyllithium

I (1.5 g, 9.1 mmol), THF (40 mL) and 30  $\mu$ L of HMPA were placed in a flame dried 100 mL Schlenk flask equipped with a Teflon covered magnetic stirring bar. The flask was sealed with a rubber septum and was cooled to  $-78^{\circ}\text{C}$  in a dry-ice/acetone bath. *n*-Butyllithium (2.5 M, 100 L, 0.25 mmol) was added slowly to the well stirred reaction mixture which became yellowish and milky. The reaction was stirred for 3 h. It was quenched by addition of 10 mL of saturated aqueous ammonium chloride while the reaction was maintained at  $-78^{\circ}\text{C}$ . Ether (300 mL) was added to dissolve the product. The organic layer was washed with water (3 x 50 mL), dried over anhydrous calcium chloride, filtered and the volatile organic solvents were removed by evaporation under reduced pressure. In this way, 1.45 g, 97% yield, of II was obtained. It was recrystallized from hot THF, mp 160-162  $^{\circ}\text{C}$ . It had the following spectral properties.  $^1\text{H}$  NMR  $\delta$ : 1.38(d, 8H,  $J = 0.9$  Hz), 1.56(d, 8H,  $J = 6.6$  Hz), 1.69(s, 12H) 5.30(d of d, 4H,  $J = 6.8$  and 0.9 Hz).  $^{13}\text{C}$

$\text{NMR} \delta$  : 14.06, 19.34, 22.99, 122.92, 130.58.  $^{29}\text{Si NMR} \delta$  : 11.87. IR (KBr)  $\nu$ : 2996, 2973, 2943, 2909, 2884, 2869, 2775, 1635, 1439, 1408, 1393, 1375, 1366, 1268, 1178, 1151, 1124, 1055, 1025, 986, 932, 766, 730, 694, 685, 672, 636  $\text{cm}^{-1}$ . GC/MS m/e (rel. intensity) 330(3.9), 329(11.7), 328(40.7)  $\text{M}^+$ , 274(2.7), 273(6.7), 272(4.2), 246(2.5), 245(4.9), 244(2.6), 218(1.0), 166(4.0), 165(11.2), 164(62.7)  $\text{C}_{10}\text{H}_{16}\text{Si}^+$ , 163(3.6), 151(1.5), 150(2.6), 149(7.2), 147(1.5), 138(3.6), 137(15.1), 136(12.0), 135(5.8), 125(1.9), 124(3.5), 123(13.5), 122(17.4), 121(7.2), 113(1.1), 112(4.9), 111(21.0), 110(100.0)  $\text{C}_6\text{H}_{10}\text{Si}^+$ , 109(37.5), 108(11.0), 97(4.7), 96(6.1), 95(47.9), 94(3.9), 84(2.4), 83(12.7), 82(15.4), 71(3.3), 70(2.5), 69(14.2), 68(3.1), 67(10.2), 58(1.5), 57(1.3), 56(0.6), 55(12.2), 54(3.1). High resolution MS m/e calcd. for  $\text{C}_{20}\text{H}_{32}\text{Si}_2$   $\text{M}^+$ : 328.2042. Found: 328.2036. Elemental Anal. Calcd. for  $\text{C}_{20}\text{H}_{32}\text{Si}_2$ : C, 73.09; H, 9.81. Found: C, 73.30; H, 9.98.

#### Acknowledgements:

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## SUPPLEMENTAL MATERIAL

**Table 1: Summary of Crystal Data and Refinement Results for  
2,3,12,13-tetramethyl-5,10-disila[4,4,4]octadeca-2,7,12,16-tetraene**

molecular weight(g/mole)	328.20
space group	P $\bar{1}$ (No.2)
molecules per unit cell	1
a (Å)	7.826(3)
b (Å)	9.415(3)
c (Å)	7.421(3)
$\alpha$ (deg)	94.11(3)
$\beta$ (deg)	114.56(3)
$\gamma$ (deg)	89.34(3)
V (Å <sup>3</sup> )	496.0(3)
crystal Dimensions (mm)	1.0x0.6x0.4
calculated density (g cm <sup>-3</sup> )	1.10
linear abs. coeff. (cm <sup>-1</sup> )	1.42
wavelength (Å) used for data collection	0.71069
$\sin\theta/\lambda$ limit (Å <sup>-1</sup> )	0.5947
total number of reflections measured	1809
number of reflections used in the structural analysis $ > 3\sigma(I) $	1478
number of variable parameters	164
final agreement factors	$R(F) = 0.0331$ $R(wF) = 0.0331$

**Table 2: Final Atomic Coordinates for  
2,3,12,13-tetramethyl-5,10-disila[4,4,4]octadeca-2,7,12,16-tetraene**

Atom	x	y	z
Si5	0.2718( 1)	0.1925( 1)	0.1046( 1)
C 1	0.5295( 3)	0.1823( 2)	0.1654( 4)
C 2	0.6007( 3)	0.3338( 2)	0.2349( 3)
C 3	0.4776( 3)	0.4342( 2)	0.2257( 3)
C 4	0.2721( 3)	0.3908( 2)	0.1493( 4)
C 6	0.2106( 3)	0.0975( 2)	0.2840( 4)
C 7	0.2255( 3)	-0.0600( 2)	0.2658( 3)
C 8	0.0879( 3)	-0.1563( 2)	0.2130( 3)
C 9	0.1157( 3)	0.1295( 3)	-0.1585( 4)
C10	0.8087( 4)	0.3575( 3)	0.3070( 5)
C11	0.5233( 4)	0.5891( 3)	0.2839( 4)
H1A	0.5539(29)	0.1500(22)	0.0563(33)
H1B	0.5893(29)	0.1212(23)	0.2604(33)
H4A	0.2017(30)	0.4326(22)	0.0347(33)
H4B	0.2228(29)	0.4178(22)	0.2407(33)
H6A	0.2929(30)	0.1346(22)	0.4077(34)
H6B	0.0891(31)	0.1256(22)	0.2689(31)
H7	0.3457(30)	-0.0936(22)	0.2945(31)
H8	0.1190(28)	-0.2521(23)	0.2047(31)
H9A	0.1371(28)	0.0324(24)	-0.1787(31)
H9B	0.1554(29)	0.1756(23)	-0.2340(32)
H10A	0.8506(30)	0.3342(22)	0.2050(33)
H10B	0.8795(30)	0.3018(23)	0.4060(33)
H10C	0.8473(29)	0.4569(24)	0.3444(32)
H11A	0.4672(29)	0.6450(23)	0.1794(33)
H11B	0.4812(29)	0.6236(22)	0.3830(33)
H11C	0.6522(31)	0.6095(22)	0.3408(32)

Table 3: Temperature Factors for  
2,3,12,13-tetramethyl-5,10-disila[4,4,4]octadeca-2,7,12,16-tetraene

Atom	$U_{11} \times 10^3$	$U_{22} \times 10^3$	$U_{33} \times 10^3$	$U_{12} \times 10^3$	$U_{13} \times 10^3$	$U_{23} \times 10^3$
Si5	400( 3)	385( 3)	581( 4)	-34( 2)	155( 3)	35( 3)
C 1	460(12)	444(12)	628(15)	40(10)	197(11)	68(11)
C 2	432(11)	524(12)	405(11)	-79( 9)	157( 9)	29( 9)
C 3	529(12)	429(11)	401(11)	-89( 9)	196(10)	18( 9)
C 4	478(13)	443(13)	673(16)	19(10)	189(12)	55(11)
C 6	488(13)	511(13)	519(13)	-60(10)	162(11)	-30(10)
C 7	431(12)	510(13)	478(13)	27(10)	109(10)	107(10)
C 8	528(13)	434(12)	486(12)	11(10)	133(10)	110(10)
C 9	537(14)	550(14)	567(14)	-73(11)	232(11)	68(11)
C10	488(15)	791(20)	762(19)	-110(13)	206(13)	59(15)
C11	798(18)	469(13)	579(15)	-134(12)	322(14)	-35(11)

The complete temperature factor is  $\exp[-2\pi^2(U_{11}h^2a^*{}^2 + U_{22}k^2b^*{}^2 + U_{33}l^2c^*{}^2 + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$

**Table 4: Bond Distances(Å)for  
2,3,12,13-tetramethyl-5,10-disila[4,4,4,4]octadeca-2,7,12,16-tetraene**

---

Si5 ---C 1	1.878( 2)
Si5 ---C 4	1.872( 2)
Si5 ---C 6	1.874( 2)
Si5 ---C 9	1.877( 2)
C 2 ---C 1	1.512( 3)
C 3 ---C 2	1.327( 3)
C 3 ---C 4	1.516( 3)
C 6 ---C 7	1.487( 3)
C 7 ---C 8	1.325( 3)
C 8 ---C 9	1.495( 3)
C 2 ---C10	1.501( 3)
C 3 ---C11	1.495( 3)
C 1 ---H1A	0.937(22)
C 1 ---H1B	0.908(22)
C 4 ---H4A	0.914(22)
C 4 ---H4B	0.930(22)
C 6 ---H6A	0.921(22)
C 6 ---H6B	0.947(21)
C 7 ---H7	0.931(21)
C 8 ---H8	0.937(21)
C 9 ---H9A	0.940(21)
C 9 ---H9B	0.884(22)
C10 ---H10A	0.953(22)
C10 ---H10B	0.912(22)
C10 ---H10C	0.972(21)
C11 ---H11A	0.917(22)
C11 ---H11B	0.959(22)
C11 ---H11C	0.934(21)

---

**Table 5: Bond Angles (deg) for  
2,3,12,13-tetramethyl-5,10-disila[4,4,4,4]octadeca-2,7,12,16-tetraene**

C 1 -Si5 -C 4	95.2( 1)
C 1 -Si5 -C 9	113.9( 1)
C 6 -Si5 -C 1	111.4( 1)
C 6 -Si5 -C 4	112.3( 1)
C 6 -Si5 -C 9	110.8( 1)
C 9 -Si5 -C 4	112.4( 1)
 Si5 -C 1 -C 2	103.7( 1)
Si5 -C 4 -C 3	103.9( 1)
Si5 -C 6 -C 7	114.2( 2)
 C 1 -C 2 -C10	116.0( 2)
C 2 -C 3 -C 4	118.2( 2)
C 2 -C 3 -C11	125.6( 2)
C 3 -C 2 -C 1	118.6( 2)
C 3 -C 2 -C10	125.4( 2)
C 4 -C 3 -C11	116.2( 2)
C 6 -C 7 -C 8	127.4( 2)
 Si5 -C 1 -H1A	113.1(13)
Si5 -C 1 -H1B	112.4(13)
Si5 -C 4 -H4A	111.3(13)
Si5 -C 4 -H4B	111.8(13)
Si5 -C 6 -H6A	104.9(13)
Si5 -C 6 -H6B	108.0(13)
Si5 -C 9 -H9A	108.8(13)
Si5 -C 9 -H9B	105.9(14)
 C 2 -C 1 -H1A	110.8(13)
C 2 -C 1 -H1B	111.7(13)
C 2 -C10 -H10A	112.0(13)
C 2 -C10 -H10B	114.0(14)
C 2 -C10 -H10C	113.1(13)
C 3 -C 4 -H4A	110.9(13)
C 3 -C 4 -H4B	111.7(13)
C 3 -C11 -H11A	112.7(14)
C 3 -C11 -H11B	111.8(13)
C 3 -C11 -H11C	113.1(13)
C 6 -C 7 -H7	115.5(13)
C 7 -C 8 -H8	117.4(13)

H6A -C 6 -C 7	110.7(13)
H6B -C 6 -C 7	112.1(13)
H7 -C 7 -C 8	117.1(13)
H1A -C 1 -H1B	105.4(18)
H4A -C 4 -H4B	107.3(19)
H6A -C 6 -H6B	106.4(18)
H9A -C 9 -H9B	106.6(19)
H10A-C10 -H10B	103.7(19)
H10A-C10 -H10C	102.5(18)
H10B-C10 -H10C	110.5(19)
H11A-C11 -H11B	105.8(19)
H11A-C11 -H11C	108.2(19)
H11B-C11 -H11C	104.6(18)

## OBSERVED AND CALCULATED STRUCTURE FACTORS FOR S1-Park2 P1BAR

PAGE 1

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC											
1	0	0	-204	1	3	0	66	-62	-5	7	0	28	-29	-3	-9	1	56	-56	1	-5	1	69	-67		
2	0	0	-204	2	3	0	24	-19	-3	7	0	93	-93	-2	-9	1	16	14	3	-5	1	86	85		
3	0	0	257	-248	3	3	0	86	-91	-2	7	0	98	96	0	-9	1	81	-78	4	-5	1	34	-32	
4	0	0	171	176	4	3	0	40	-41	0	7	0	28	-31	2	-9	1	54	54	5	-5	1	102	-105	
5	0	0	80	-89	5	3	0	139	143	1	7	0	10	14	3	-9	1	30	29	6	-5	1	19	20	
6	0	0	80	-77	6	3	0	64	63	2	7	0	142	148	4	-9	1	36	-36	7	-5	1	58	57	
7	0	0	17	13	7	3	0	52	-54	3	7	0	33	33	-6	-8	1	37	-35	-8	-4	1	11	13	
8	0	0	32	30	-7	4	0	48	49	4	7	0	91	-93	-3	-8	1	54	56	-7	-4	1	64	-64	
-8	0	0	50	47	-6	4	0	45	45	5	7	0	46	-50	-2	-8	1	27	27	-6	-4	1	19	-19	
-7	-1	0	41	-40	-5	4	0	65	-63	6	7	0	46	46	-1	-8	1	33	-31	-5	-4	1	116	116	
-6	-1	0	126	-128	-3	4	0	200	209	-6	8	0	17	14	0	-8	1	136	-138	-4	-4	1	122	-122	
-5	-1	0	64	57	-2	4	0	104	98	-5	8	0	14	8	1	-8	1	63	62	-3	-4	1	68	-71	
-4	-1	0	100	104	-1	4	0	247	-249	-4	8	0	86	-86	2	-9	1	52	54	-2	-4	1	25	30	
-3	-1	0	233	-223	0	4	0	356	-348	-3	8	0	11	15	3	-8	1	33	-33	-1	-4	1	197	197	
-2	-1	0	190	-194	1	4	0	286	277	-1	8	0	60	56	4	-8	1	24	-23	0	-4	1	121	-118	
0	0	1	0	50	51	2	4	0	90	-87	0	8	0	101	-103	5	-8	1	22	19	1	-4	1	51	-55
-1	-1	0	109	107	3	4	0	60	-58	-1	8	0	72	71	-6	-7	1	42	40	2	-4	1	103	106	
2	0	0	158	-155	4	4	0	16	16	2	8	0	65	61	-5	-7	1	55	-56	3	-4	1	62	62	
3	-1	0	304	299	5	4	0	78	83	3	8	0	24	-22	-4	-7	1	54	-56	4	-4	1	115	-118	
4	-1	0	128	-131	6	4	0	29	-31	5	8	0	39	38	-3	-7	1	81	82	5	-4	1	44	45	
5	-1	0	82	-80	7	4	0	26	-22	-4	9	0	43	-40	-2	-7	1	87	91	6	-4	1	47	47	
6	-1	0	27	27	-7	5	0	64	62	-3	9	0	46	45	-1	-7	1	138	-143	-8	-3	1	47	-47	
7	-1	0	35	32	-6	5	0	17	-16	-2	9	0	32	31	0	-7	1	38	37	-7	-3	1	26	-22	
8	-1	0	34	-32	-5	5	0	96	-93	-1	9	0	82	-84	1	-7	1	32	33	-6	-3	1	35	33	
-8	2	0	35	32	-4	5	0	92	89	0	9	0	20	-17	2	-7	1	53	54	-5	-3	1	99	103	
-7	2	0	79	-80	-3	5	0	117	118	1	9	0	71	67	3	-7	1	124	-125	-4	-3	1	60	-65	
-6	2	0	61	56	-2	5	0	73	-76	3	9	0	63	-65	5	-7	1	20	18	-3	-3	1	32	-28	
-5	-2	0	13	16	-1	5	0	141	-138	4	9	0	15	-15	-7	-6	1	54	53	-2	-3	1	17	-18	
-4	2	0	14	-13	0	5	0	193	192	-3	10	0	45	46	-6	-6	1	61	-62	-1	-3	1	128	-121	
-3	2	0	157	-154	1	5	0	114	-112	-2	10	0	25	-25	-5	-6	1	110	-110	0	-3	1	240	-241	
-2	2	0	156	154	3	5	0	93	-89	-1	10	0	33	-35	-4	-6	1	86	87	1	-3	1	167	173	
-1	2	0	173	175	4	5	0	202	207	0	10	0	44	39	-3	-6	1	128	131	2	-3	1	56	59	
0	2	0	118	-120	5	5	0	105	-109	2	10	0	74	-72	-2	-6	1	37	-40	3	-3	1	21	12	
-1	2	0	200	-204	6	5	0	36	-32	3	10	0	19	-15	-1	-6	1	132	-137	4	-3	1	49	-48	
2	2	0	58	-59	7	5	0	12	14	-1	11	0	50	46	0	-6	1	136	134	5	-3	1	74	73	
-8	3	0	34	-35	-1	6	0	67	76	-3	10	1	48	-48	-7	-5	1	33	-30	-5	-2	1	8	2	
4	2	0	8	5	-7	6	0	61	-63	1	11	0	62	-61	2	-6	1	85	-86	-4	-2	1	113	-110	
5	2	0	92	93	-6	6	0	205	-109	2	10	0	74	-72	-2	-6	1	103	-101	7	-3	1	40	-39	
6	2	0	34	33	-4	6	0	53	53	0	-11	1	49	45	3	-6	1	47	-47	-8	-2	1	69	-68	
7	2	0	13	-13	-3	6	0	30	-35	-1	-11	1	15	13	5	-6	1	23	-23	-7	-2	1	43	40	
8	2	0	54	-52	-2	6	0	67	-63	-4	-10	1	34	32	6	-6	1	28	-25	-6	-2	1	126	129	
-8	3	0	34	-35	-1	6	0	67	76	-3	-10	1	48	-48	-7	-5	1	85	-86	-4	-2	1	113	-110	
4	2	0	8	5	-7	6	0	61	-63	1	11	0	62	-61	2	-6	1	81	-84	-3	-2	1	67	61	
-6	3	0	17	18	0	6	0	48	53	-2	-10	1	49	-48	-6	-5	1	49	46	-4	-2	1	121	121	
-5	3	0	129	127	1	6	0	27	-29	-1	-10	1	43	45	3	-6	1	47	-47	-8	-2	1	139	-145	
-4	3	0	121	-114	2	6	0	44	43	0	-10	1	25	22	-4	-5	1	149	153	-2	-2	1	126	129	
-3	3	0	87	78	3	6	0	126	123	1	-10	1	59	-59	-3	-5	1	43	-42	1	-2	1	109	112	
-2	3	0	18	25	4	6	0	118	-120	3	-10	1	71	70	-2	-5	1	43	-42	2	-2	1	109	112	
-1	3	0	7	-4	5	6	0	56	-55	-5	-9	1	62	-63	-1	-5	1	124	129	3	-2	1	48	-46	
0	3	0	220	-213	7	6	0	59	60	-4	-9	1	27	-23	0	-5	1	76	79	4	-2	1	157	160	

## OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Si-Park2 PIBAR

PAGE 2

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
5	-2	1	33	28	-5	2	1	73	75	0	5	1	195	201	3	9	1	39	-37
7	-2	1	48	-47	-4	2	1	242	-243	1	5	1	112	-113	-4	10	1	49	46
-8	-1	1	35	-34	-3	2	1	53	-52	2	5	1	139	-143	-2	10	1	37	38
-7	-1	1	72	71	-2	2	1	29	-34	3	5	1	47	-45	-2	10	1	49	-49
-6	-1	1	82	84	-1	2	1	299	312	4	5	1	49	49	-1	10	1	11	-10
-5	-1	1	25	-29	0	2	1	22	21	6	5	1	10	-7	0	10	1	46	47
-4	-1	1	17	16	1	2	1	146	149	-7	6	1	51	-49	1	10	1	24	-23
-3	-1	1	49	49	2	2	1	68	65	-6	6	1	32	-33	2	10	1	53	-51
-2	-1	1	122	-124	3	2	1	49	-45	-5	6	1	63	65	-1	11	2	38	39
-1	-1	1	209	-210	4	2	1	110	-112	-4	6	1	121	121	0	11	2	40	41
1	-1	1	188	-185	5	2	1	43	48	-3	6	1	107	-114	-3	10	2	43	-45
2	-1	1	151	-151	7	2	1	25	-24	-2	2	1	20	18	-1	10	2	71	70
3	-1	1	77	-75	-8	3	1	37	-36	-1	6	1	13	-13	0	10	2	10	-10
4	-1	1	184	179	-7	3	1	17	19	0	6	1	17	14	1	10	2	44	-40
5	-1	1	18	-18	-5	3	1	26	-23	1	6	1	84	-81	2	10	2	16	18
6	-1	1	109	-108	-4	3	1	116	-121	2	6	1	46	52	-5	9	2	22	25
7	-1	1	22	21	-3	3	1	85	86	3	6	1	5	56	-4	9	2	30	-32
-8	0	1	34	29	-2	3	1	137	136	4	6	1	1	-73	-2	10	2	19	14
-7	0	1	13	18	-1	3	1	165	-170	5	6	1	24	-23	0	9	2	95	-95
-6	0	1	80	82	0	3	1	183	179	6	6	1	44	48	1	9	2	53	55
-5	0	1	29	32	-1	3	1	155	154	-7	7	1	27	-30	2	9	2	39	37
-4	0	1	232	230	2	3	1	42	48	-5	7	1	43	43	4	9	2	18	-14
-3	0	1	282	-274	3	3	1	21	-25	-4	7	1	34	-32	-6	8	2	48	51
-2	0	1	56	-58	4	3	1	19	-18	-3	7	1	16	-16	-1	8	2	113	-112
-1	0	1	201	207	5	3	1	54	54	-2	7	1	12	-9	0	8	2	19	20
0	1	1	264	-260	7	3	1	36	-31	1	7	1	100	98	2	8	2	60	61
1	0	1	131	-128	-8	4	1	18	-14	0	7	1	78	-80	3	8	2	64	-67
2	0	1	271	269	-7	4	1	51	47	1	7	1	75	78	4	8	2	54	56
3	0	1	21	-20	-6	4	1	26	26	2	7	1	9	8	-7	7	2	47	47
4	0	1	176	-185	-5	4	1	108	-113	3	7	1	24	-26	-4	7	2	26	26
5	0	1	37	-36	-4	4	1	63	67	4	7	1	45	-45	-3	7	2	61	64
6	0	1	75	72	-3	4	1	160	171	5	7	1	35	35	-2	7	2	106	-104
7	0	1	34	-34	-2	4	1	85	-87	-4	8	1	62	-65	-1	7	2	136	-138
8	-1	1	74	-76	-1	4	1	230	-231	-2	8	1	26	-26	0	7	2	62	58
9	-1	1	34	34	0	4	1	77	74	-1	8	1	28	-28	1	7	2	104	107
10	-1	1	120	-105	1	4	1	25	22	0	8	1	10	17	2	7	2	80	-82
11	-1	1	273	-270	2	4	1	104	-106	-1	8	1	88	89	3	7	2	42	-42
12	-1	1	283	293	3	4	1	138	-137	2	8	1	49	47	4	7	2	40	40
13	0	1	200	-197	4	4	1	210	211	3	8	1	74	-75	5	7	2	51	53
14	0	1	190	-199	5	4	1	20	20	4	8	1	32	-32	-6	6	2	102	-27
15	1	1	105	-103	-5	5	1	195	181	-3	9	1	46	42	-5	6	2	112	-112
16	1	1	116	115	6	4	1	25	-25	5	8	1	48	-48	-5	6	2	99	102
17	1	1	164	162	7	4	1	15	-13	-5	9	1	43	-44	-4	6	2	102	-102
18	1	1	283	293	3	4	1	138	-137	2	8	1	49	47	-3	6	2	42	-41
19	1	1	200	-197	4	4	1	210	211	3	8	1	74	-75	0	3	2	53	53
20	1	1	190	-199	5	4	1	230	-231	-2	8	1	26	-26	-6	6	2	116	120
21	1	1	120	-105	1	4	1	25	22	0	8	1	10	17	2	7	2	80	-82
22	1	1	273	-270	2	4	1	104	-106	-1	8	1	88	89	3	7	2	42	-42
23	1	1	283	293	3	4	1	138	-137	2	8	1	49	47	-3	6	2	42	-41
24	1	1	200	-197	4	4	1	210	211	3	8	1	74	-75	0	3	2	53	53
25	1	1	190	-199	5	4	1	230	-231	-2	8	1	26	-26	-6	6	2	116	120
26	1	1	120	-105	1	4	1	25	22	0	8	1	10	17	2	7	2	80	-82
27	1	1	273	-270	2	4	1	104	-106	-1	8	1	88	89	3	7	2	42	-42
28	1	1	283	293	3	4	1	138	-137	2	8	1	49	47	-3	6	2	42	-41
29	1	1	200	-197	4	4	1	210	211	3	8	1	74	-75	0	3	2	53	53
30	1	1	190	-199	5	4	1	230	-231	-2	8	1	26	-26	-6	6	2	116	120
31	1	1	105	-103	-5	5	1	195	181	-3	9	1	46	42	-1	6	2	116	120
32	1	1	116	115	6	4	1	25	-25	5	8	1	48	-48	-5	6	2	116	120
33	1	1	164	162	7	4	1	15	-13	-5	9	1	43	-44	-4	6	2	213	212
34	1	1	122	-124	-6	5	1	76	-78	-4	9	1	35	-31	-3	6	2	42	-41
35	1	1	105	-103	-5	5	1	33	34	-3	9	1	46	42	-2	6	2	116	120
36	1	1	116	115	6	4	1	20	20	4	8	1	10	17	2	7	2	80	-82
37	1	1	164	162	7	4	1	15	-13	-5	9	1	43	-44	-4	6	2	213	212
38	1	1	283	293	3	4	1	138	-137	2	8	1	49	47	-3	6	2	42	-41
39	1	1	200	-197	4	4	1	210	211	3	8	1	74	-75	0	3	2	53	53
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41	1	1	105	-103	-5	5	1	195	181	-3	9	1	46	42	-1	6	2	116	120
42	1	1	116	115	6	4	1	25	-25	5	8	1	48	-48	-5	6	2	213	212
43	1	1	164	162	7	4	1	15	-13	-5	9	1	43	-44	-4	6	2	213	212
44	1	1	122	-124	-6	5	1	76	-78	-4	9	1	35	-31	-3	6	2	42	-41
45	1	1	105	-103	-5	5	1	33	34	-3	9	1	46	42	-2	6	2	116	120
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48	1	1	283	293	3	4	1	138	-137	2	8	1	49	47	-3	6	2	42	-41
49	1	1	200	-197	4	4	1	210	211	3	8	1	74	-75	0	3	2	53	53
50	1	1	190	-199	5	4	1	230	-231	-2	8	1	26	-26	-6	6	2	116	120
51	1	1	105	-103	-5	5	1	195	181	-3	9	1	46	42	-1	6	2	116	120
52	1	1	116	115	6	4	1	25	-25	5	8	1	48	-48	-5	6	2	213	212
53	1	1	164	162	7	4	1	15	-13	-5	9	1	43	-44	-4	6	2	213	212
54	1	1</td																	

## OBSERVED AND CALCULATED STRUCTURE FACTORS FOR SI-Park2 P1BAR

PAGE 3

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	
6	-3	2	29	30	-2	1	2	155	152	4	2	143	144	-5	9	2	51	-48		
7	-3	2	54	-54	-1	1	2	11	-8	5	4	2	33	-34	-4	9	2	15	-132	
-8	-2	2	35	-30	0	1	2	160	-155	6	4	2	32	-31	-3	9	2	28	135	
-7	-2	2	67	63	2	1	2	242	235	-8	5	2	42	40	-2	9	2	38	136	
-6	-2	2	81	83	3	1	2	63	-60	-7	5	2	28	-26	-1	9	2	25	138	
-5	-2	2	88	90	4	1	2	146	-147	-6	5	2	66	-62	0	9	2	77	145	
-4	-2	2	158	-156	5	1	2	14	12	-5	5	2	79	77	1	9	2	27	146	
-3	-2	2	198	194	6	1	2	48	45	-4	5	2	88	89	2	9	2	38	147	
-2	-2	2	27	30	-8	2	2	55	-55	-3	5	2	112	-116	3	9	2	15	148	
-1	-2	2	143	-135	-7	2	2	30	29	-2	2	102	-102	-3	10	2	26	149		
0	-2	2	130	-128	-6	2	2	35	-33	-1	5	2	149	156	-2	10	2	49	150	
2	-2	2	26	-28	-5	2	2	38	41	1	5	2	131	-131	-1	10	2	32	151	
3	-2	2	55	-60	-4	2	2	183	-179	2	5	2	106	-103	0	10	2	45	152	
4	-2	2	82	84	-3	2	2	133	124	3	2	190	192	1	10	2	21	153		
5	-2	2	86	86	-2	2	2	52	44	4	5	2	39	-39	-4	10	2	12	154	
6	-2	2	48	-48	-1	2	2	36	-35	5	5	2	12	-11	-3	10	2	21	155	
-8	-1	2	41	42	0	2	2	48	-47	-7	6	2	52	-53	-2	10	2	55	156	
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-6	-1	2	28	-22	2	2	2	150	151	-5	6	2	82	84	0	10	3	50	158	
-5	-1	2	116	-121	3	2	2	76	-75	-4	6	2	10	14	-1	10	3	14	159	
-4	-1	2	304	4	2	123	-121	-3	2	137	-130	-4	9	3	57	-55	-8	4	35	
-3	-1	2	10	11	5	2	2	12	-10	-2	6	2	78	83	-3	9	3	28	160	
-2	-1	2	26	21	6	2	2	25	26	-1	6	2	74	72	-2	9	3	51	161	
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2	-1	2	27	24	-7	3	2	23	22	2	6	2	26	26	1	9	3	17	164	
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-3	0	2	32	4	3	2	34	32	4	3	2	34	-32	-7	3	3	34	174		
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-1	0	2	117	-120	6	3	2	46	-45	4	7	2	12	-10	-4	7	3	46	176	
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-7	-1	2	73	-72	-1	4	2	76	-73	-2	8	2	64	62	3	7	3	59	182	
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-5	-1	2	67	68	1	4	2	102	-97	-1	8	2	39	-36	-5	6	3	39	184	
-4	-1	2	90	-85	2	4	2	60	-64	3	8	2	26	-26	-4	6	3	62	185	
-3	1	2	79	-83	3	4	2	78	-80	4	8	2	15	13	-3	6	3	123	186	

## OBSERVED AND CALCULATED STRUCTURE FACTORS FOR SI-Park2 P1BAR

PAGE 4

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	
6	-3	2	29	30	-2	1	2	155	152	4	2	143	144	-5	9	2	51	-48		
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-8	-2	2	35	-30	0	1	2	160	-155	6	4	2	32	-31	-3	9	2	28	135	
-7	-2	2	67	63	2	1	2	242	235	-8	5	2	42	40	-2	9	2	38	36	
-6	-2	2	81	83	3	1	2	63	-60	-7	5	2	28	-26	-1	9	2	19	138	
-5	-2	2	88	90	4	1	2	146	-147	-6	5	2	66	-62	0	9	2	77	145	
-4	-2	2	158	-156	5	1	2	14	12	-5	5	2	79	77	1	9	2	27	50	
-3	-2	2	198	194	6	1	2	48	45	-4	5	2	88	89	2	9	2	41	50	
-2	-2	2	27	30	-8	2	2	55	-55	-3	5	2	112	-116	3	9	2	15	36	
-1	-2	2	143	-135	-7	2	2	30	29	-2	2	102	-102	-3	10	2	26	150		
0	-2	2	130	-128	-6	2	2	35	-33	-1	5	2	149	156	-2	10	2	49	-47	
2	-2	2	26	-28	-5	2	2	38	41	1	5	2	131	-131	-1	10	2	32	34	
3	-2	2	55	-60	-4	2	2	183	-179	2	5	2	106	-103	0	10	2	45	46	
4	-2	2	82	84	-3	2	2	133	124	3	2	190	192	1	10	2	21	21		
5	-2	2	86	86	-2	2	2	52	44	4	5	2	39	-39	-4	10	2	12	135	
6	-2	2	48	-48	-1	2	2	36	-35	5	5	2	12	-11	-3	10	2	21	-80	
-8	-1	2	41	42	0	2	2	48	-47	-7	6	2	52	-53	-2	10	2	55	-80	
-7	-1	2	83	85	1	2	2	219	213	-6	6	2	54	55	-1	10	3	47	40	
-6	-1	2	28	-22	2	2	2	150	151	-5	6	2	82	84	0	10	3	50	46	
-5	-1	2	116	-121	3	2	2	76	-75	-4	6	2	10	14	-1	10	3	15	74	
-4	-1	2	304	4	2	123	-121	-3	2	137	-130	-4	9	3	57	-55	-8	39	159	
-3	-1	2	10	11	5	2	2	12	-10	-2	6	2	78	83	-3	9	3	28	160	
-2	-1	2	26	21	6	2	2	25	26	-1	6	2	74	72	-2	9	3	51	161	
0	0	1	2	196	196	7	2	2	32	-33	0	6	2	101	-106	-1	9	3	36	162
1	-1	2	178	-166	-8	3	2	36	-36	1	6	2	122	-125	0	9	3	28	163	
2	-1	2	27	24	-7	3	2	23	22	2	6	2	26	26	1	9	3	17	164	
3	-1	2	88	88	-6	3	2	58	57	3	2	27	28	2	9	3	17	165		
4	-1	2	103	104	-5	3	2	153	-148	4	6	2	14	15	3	9</				

## OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Si-Park2 PIBAR

PAGE 5

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
2	1	3	106	110	-6	5	3	14	-12	-2	-10	4	40	40
-4	-2	3	112	71	-67	-5	5	13	-10	-1	-10	4	35	36
-3	-2	3	180	176	4	1	3	53	-53	-4	-16	0	99	98
-2	-2	3	50	-57	5	1	3	78	-79	-3	-16	0	-22	-22
-1	-2	3	253	-243	6	1	3	45	44	-2	-5	0	79	-77
0	-2	3	290	289	-8	2	3	46	-46	-1	-5	0	4	4
1	-2	3	111	109	-7	2	3	45	42	0	5	3	-4	4
2	-2	3	87	-85	-6	2	3	63	64	1	5	3	-4	4
3	-2	3	71	-78	-5	2	3	40	-34	2	5	3	-4	4
4	-2	3	22	24	-4	2	3	19	-26	3	5	3	-4	4
5	-2	3	28	27	-3	2	3	25	23	4	5	3	-4	4
6	-2	3	41	-39	-2	2	3	121	125	5	5	3	-4	4
-8	-1	3	82	79	-1	2	3	128	-123	-7	6	3	-5	3
-7	-1	3	19	22	0	2	3	59	-58	-6	6	3	-5	3
-6	-1	3	146	-147	1	2	3	119	120	-5	6	3	-5	3
-5	-1	3	80	-84	2	2	3	22	-19	-4	6	3	-5	3
-4	-1	3	249	255	3	2	3	62	-81	-3	6	3	-5	3
-3	-1	3	13	-15	4	2	3	19	16	-2	6	3	-5	3
-2	-1	3	213	-205	5	2	3	29	29	-1	6	3	-5	3
0	-1	3	67	62	-8	3	3	20	-18	0	6	3	-5	3
1	-1	3	16	-15	-7	3	3	30	30	-1	6	3	-5	3
2	-1	3	100	-105	-6	3	3	50	52	2	6	3	-5	3
3	-1	3	135	139	-5	3	3	108	-106	4	6	3	-5	3
4	-1	3	31	34	-3	3	3	75	77	-6	7	3	-5	3
5	-1	3	44	-45	-2	3	3	19	-24	-4	7	3	-5	3
-8	0	3	53	53	-1	3	3	68	-71	-3	7	3	-5	3
-7	0	3	89	-88	0	3	3	179	177	-2	7	3	-5	3
-5	0	3	173	178	1	3	3	70	72	-1	7	3	-5	3
-4	0	3	37	32	2	3	3	35	-33	0	7	3	-5	3
-3	0	3	108	-107	3	3	3	121	-124	-4	7	3	-5	3
-2	0	3	228	-227	4	3	3	72	71	2	7	3	-5	3
-1	0	3	104	100	5	3	3	13	-12	3	7	3	-5	3
0	0	3	162	-149	6	3	3	39	-39	-6	8	3	-5	3
1	0	3	61	-63	-8	4	3	46	42	4	8	3	-5	3
2	0	3	87	84	-7	4	3	30	32	-3	8	3	-5	3
3	0	3	38	42	-6	4	3	86	-86	-1	8	3	-5	3
4	0	3	42	-44	-5	4	3	61	-58	1	8	3	-5	3
5	0	3	48	49	-4	4	3	57	61	2	8	3	-5	3
6	0	3	59	58	-2	4	3	51	-52	3	8	3	-5	3
-8	1	3	32	-28	-1	4	3	64	63	-5	9	3	-2	1
-7	1	3	14	-14	0	4	3	147	148	-4	9	3	-1	1
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-4	1	3	47	-42	2	4	3	67	-71	0	9	3	-1	1
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0	1	3	155	-161	-8	5	3	27	24	-1	10	3	-1	1
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## OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Si-Park2 PIBAR

M	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC				
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0	1	0	70	-73	-2	4	72	-2	-9	5	15	17	3	-4	4	0	4	0	5				
1	2	0	90	-19	0	4	127	-1	-9	5	63	4	4	-4	-4	1	5	18	-17				
2	3	0	22	-30	-32	1	4	24	-19	0	-9	5	16	-8	-5	12	15	-6	1				
3	4	0	4	30	-34	2	4	5	11	-6	-8	5	22	-7	-3	3	28	40	47				
4	5	0	34	-75	3	4	59	-60	-5	-8	5	50	-53	-6	-3	5	21	-5	-1				
5	6	-8	1	4	-6	4	110	108	-3	-8	5	70	69	-5	-3	5	55	-3	1				
6	7	-7	1	4	50	4	60	-2	-8	5	34	-31	-4	-4	3	5	16	-2	1				
7	8	-6	1	4	31	5	4	14	-1	-8	5	26	-2	-3	5	23	-27	0	1				
8	9	-5	1	4	49	45	-7	5	4	48	-51	-6	68	-1	-3	5	99	-104	-1	1			
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10	11	-3	1	4	37	42	-4	5	4	18	-21	-4	-7	5	73	70	-1	-3	5	22	3	1	
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19	20	-6	2	4	124	124	-4	6	4	94	-95	-5	-6	5	74	72	-3	-2	5	45	-50	4	
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23	24	-2	2	4	112	-121	0	6	4	46	-48	0	-6	5	48	-49	-1	-2	5	51	-5	-1	
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29	30	-5	2	4	41	-5	7	4	60	-61	-5	-5	5	23	-23	-7	-1	5	67	-70	2	3	
30	31	-6	3	4	28	-30	-2	7	4	55	55	-4	-5	5	48	-43	-6	-1	5	53	-73	-6	5
31	32	-7	3	4	54	55	-1	7	4	91	-90	-3	-5	5	18	-16	-5	-1	5	105	108	4	3
32	33	-3	2	4	37	-39	3	6	4	43	-41	-7	-5	5	17	-19	4	-2	5	59	60	-2	3
33	34	-4	2	4	82	82	-6	7	4	39	38	-1	-5	5	58	-54	-2	-1	5	63	-66	-8	4
34	35	-5	2	4	41	-49	1	7	4	39	-37	0	-5	5	56	-52	-3	-1	5	45	-41	-7	4
35	36	-4	3	4	88	88	3	7	4	38	-37	0	-5	5	56	-59	-2	-1	5	70	73	-6	4
36	37	-3	3	4	97	98	-5	8	4	33	-30	1	-5	5	52	55	-1	-1	5	174	171	-5	4
37	38	-2	3	4	82	-75	-4	8	4	37	36	2	-5	5	59	-62	0	-1	5	105	108	4	3
38	39	-1	3	4	143	-147	-3	8	4	42	3	-5	5	43	-41	1	-1	5	98	-101	-3	4	
39	40	0	3	4	121	119	-2	8	4	53	-53	-8	-4	5	15	-10	2	-1	5	54	58	-2	4
40	41	-1	3	4	45	-45	-1	8	4	44	-43	-7	-4	5	23	26	3	-1	5	22	21	-1	4
41	42	2	3	4	30	-35	0	8	4	30	27	-6	-4	5	63	59	4	-1	5	71	76	0	4
42	43	3	4	47	49	1	8	4	13	16	-5	-4	5	18	-18	-8	0	5	61	-60	1	4	
43	44	4	4	47	49	-3	-4	6	59	55	0	0	6	49	-51	-2	5	6	30	29	-6	1	

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## OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Si-Park2 P1BAR

	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC				
-3	4	7	18	12	-3	-5	8	21	-6	-2	8	23	27	-6	0	8	33	33	-3	1	8	16	-16	
-2	4	7	50	51	-4	-4	8	37	-5	-2	8	19	19	-5	0	8	25	24	-2	1	8	19	-21	
-0	4	7	42	-41	-1	-4	8	13	-3	-2	8	69	74	-4	0	8	10	10	-6	2	8	16	-18	
-6	5	7	36	38	-6	-3	8	44	-4	-2	8	67	73	-2	0	8	20	22	-4	2	8	16	-21	
-5	5	7	37	-39	-5	-3	8	51	-6	-1	8	35	37	-1	0	8	24	22	-2	2	8	16	-35	
-2	5	7	37	38	-3	-3	8	17	22	-5	-1	8	13	10	0	0	8	16	-1	2	8	16	-43	
-1	5	7	55	-53	-2	-3	6	17	-22	-4	-1	8	38	-6	0	8	38	-6	1	8	16	-15		
-3	6	5	23	26	2	-4	6	31	-28	-6	-1	6	23	18	-4	6	15	22	-1	-1	7	44	-46	
-5	6	5	11	-10	3	-4	6	18	21	-5	1	6	99	-104	-3	6	6	27	-28	0	-1	7	20	-19
-2	6	5	56	55	-8	-3	6	21	18	-4	1	6	40	40	-2	6	6	31	29	-1	-1	7	32	33
-1	6	5	46	-7	-3	6	59	58	-3	1	6	74	77	-1	6	6	28	-27	2	-1	7	19	20	
0	6	5	55	-55	-5	-3	6	62	-59	-2	-1	6	15	16	0	6	6	18	-19	-6	0	7	45	44
1	6	5	30	30	-4	-3	6	63	59	-1	1	6	45	-44	-5	6	6	21	-17	-5	0	7	31	-33
2	6	5	12	-16	-3	6	27	-32	0	1	6	37	33	-3	7	6	30	30	-4	0	7	16	19	
-5	7	5	35	-35	-2	-3	6	49	-49	1	1	6	47	47	-2	7	6	23	-22	-3	0	7	22	27
-3	7	5	16	15	-1	-3	6	42	-41	3	1	6	43	-45	-1	7	6	15	16	-2	0	7	40	-43
-2	7	5	20	-22	0	-3	6	36	-39	-7	2	6	31	34	-4	7	7	28	-29	-1	0	7	47	-48
0	7	5	48	48	2	-3	6	12	-13	-6	-1	6	60	-58	-3	7	7	55	-55	0	0	7	17	14
0	8	5	42	41	-5	-2	6	73	73	-1	2	6	23	-19	0	6	7	26	-23	-6	1	7	20	-22
-3	8	6	22	-20	-3	-2	6	13	12	0	2	6	65	68	-5	5	7	24	-25	1	0	7	43	45
-2	8	6	55	-53	-2	-2	6	152	-138	2	2	6	25	-24	-3	5	7	60	59	-3	1	7	41	42
0	8	6	42	43	-1	-2	6	69	71	-7	3	6	10	-7	-2	5	7	25	-2	1	7	20	-19	
-6	7	6	70	-73	0	-2	6	50	-50	-6	3	6	36	-1	-5	7	24	-22	-1	1	7	24	-24	
-5	7	6	86	83	0	-2	6	13	-12	-5	3	6	39	39	-7	4	7	14	11	0	1	7	50	51
-3	7	6	20	-14	2	-2	6	23	22	-4	3	6	34	34	-6	4	7	27	-24	-7	2	7	14	16
-2	7	6	33	-31	3	-2	6	40	43	-3	3	6	49	-45	-5	4	7	34	-31	-6	2	7	41	-43
-1	7	6	58	60	-8	-1	6	26	29	-2	3	6	69	-77	-4	4	7	43	45	-5	2	7	11	-6
1	7	6	37	-38	-7	-1	6	17	-18	-1	3	6	96	93	-3	4	7	26	25	-4	2	7	31	32
-7	6	6	61	-61	-6	-1	6	23	20	0	3	6	19	-18	-1	4	7	10	9	-2	2	7	24	-24
-6	6	6	36	35	-5	-1	6	15	15	1	3	6	13	-8	0	4	7	16	-1	2	7	53	50	
-5	6	6	14	12	-4	-1	6	22	-21	2	3	6	37	-39	-7	3	7	13	-14	0	2	7	25	25
-3	6	6	49	-46	-3	-1	6	56	-56	-7	4	6	42	-42	-6	3	7	48	-46	-7	3	7	33	-33
-2	6	6	86	86	-2	-1	6	130	-132	-6	4	6	29	31	-5	3	7	16	17	-6	3	7	23	-25
-1	6	6	31	26	-1	-1	6	67	-66	-5	4	6	66	-64	-4	3	7	52	51	-5	3	7	19	18
0	6	6	28	28	0	-1	6	52	-53	-4	4	6	19	-18	-2	3	7	76	-81	-3	3	7	21	-22
-7	5	6	20	-21	-1	-1	6	29	-31	-3	4	6	70	-70	-1	3	7	38	41	-1	3	7	53	53
-6	5	6	29	29	2	-1	6	87	87	-2	4	6	23	27	0	3	7	28	24	1	3	7	30	-31
-5	5	6	11	-11	3	-1	6	13	-13	-1	4	6	14	16	-8	2	7	35	34	-5	4	7	21	19
-4	5	6	28	-26	-8	0	6	51	0	4	6	34	-31	-7	-2	7	29	-29	-4	7	65	-65		

	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC				
-3	4	7	18	12	-3	-5	8	21	20	-6	-2	8	23	27	-6	0	8	33	33	-3	1	8	16	-16
-2	4	7	50	51	-4	-4	8	37	38	-5	-2	8	19	19	-5	0	8	25	24	-2	1	8	19	-21
-0	4	7	42	-41	-1	-4	8	13	-3	-2	8	69	74	-4	0	8	10	-10	-2	2	8	16	-18	
-6	5	7	36	38	-6	-3	8	44	-47	-2	2	8	67	73	-2	0	8	20	22	-4	2	8	16	-21
-5	5	7	37	-39	-5	-3	8	51	54	-6	-1	8	35	37	-1	0	8	24	-22	-2	2	8	16	-35
-2	5	7	37	38	-3	-3	8	17	22	-5	-1	8	13	10	0	0	8	16	-18	-1	2	8	16	-43
-1	5	7	55	-53	-2	-3	8	19	-22	-4	-1	8	49	52	0	-1	8	38	-38	-6	1	8	16	-15
-3	6	7	34	33	-1	-3	8	49	52	0	-1	8	18	18	-1	0	8	18	-17	-2	3	8	16	-39

	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC				
-5	-1	7	5	6	44	-42	-4	-1	7	7	-1	7	7	44	-46	-5	1	8	16	-23				
-1	5	6	58	55	-1	5	6	44	-42	-2	2	6	66	69	-2	0	8	16	-16					
-5	6	5	19	14	-8	-1	6	40	40	-3	6	6	31	29	-3	0	8	16	-21					
-2	6	5	56	55	-2	-3	6	44	-47	-2	2	6	66	69	-2	0	8	16	-18					
-1	6	5	46	-7	-3	6	59	58	-3	1	6	74	77	-1	0	8	16	-20						
0	6	5	55	55	-5	-3	6	62	-59	-2	1	6	15	16	-2	0	8	16	-19					
1	6	5	30	30	-4	-3	6	63	59	-1	1	6	45	-44	-5	0	8	16	-15					
2	6	5	12	-16	-3	6	27	-32	0	1	6	37	33	-3	7	6	30	30	-4	0	7	16	19	
-5	7	5	35	-35	-2	-3	6	49	-49	1	1	6	47	47	-2	7	6	23	-22	-3	0	7	22	27
-3	7	5	16	15	-1	-3	6	42	-41	3	1	6	43	-45	-1	7	6	15	16	-2	0	7	40	-43
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0	8	5	48	48	2	-3	6	23	22	-4	3	6	34	34	-6	4	7	27	-24	-7	2	7	14	16
-3	8	6	22	-20	-3	2	6	13	12	0	2	6	65	68	-5	5	7	20	-18	-5	1	7	21	-21
-2	8	6	55	-53	-2	-2	6	152	-138	2	2	6	25	-24	-3	5	7	60	59	-3	1	7	41	42
0	8	6	42																					